Screening effects in ferroelectric resistive switching of BiFeO3 thin films

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Screening effects in ferroelectric resistive switching of $BiFeO_3$ thin films

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We investigate ferroelectric resistive switching in $BiFeO_3$ thin films by performing local conductivity measurements. By comparing conduction characteristics at artificially up-polarized domains with those at as-grown down-polarized domains, the change in resistance is attributed to the modification of the electronic barrier height at the interface with the electrodes, upon the reversal of the electrical polarization. We also study the effect of oxygen vacancies on the observed conduction and we propose the existence of a different screening mechanism for up and down polarized domains. [http://dx.doi.org/10.1063/1.4875355]

Using metal-ferroelectric junctions as switchable diodes was proposed several decades ago[1] and it was shown to actually work in $PbZr_{1-x}Ti_xO_3$ (PZT) by Blom *et al.*,[2] who reported switching in the rectification direction accompanied by changes of the current of about 2 orders of magnitude, upon switching the polarization direction of the ferroelectric layer. This form of resistive switching enables the read out of a ferroelectric memory state at higher speed compared to the capacitive design, without destroying the information in each reading cycle.[3–5] Apart from these examples and few others,[6] transport through ferroelectrics had not been investigated in detail until less than a decade ago. The reason for that is the large turn-on voltages that were expected for the Schottky behavior of these large band-gap semiconductors.[7] However, as the thin film deposition techniques improve and better layers with lower thicknesses are achieved, metal-ferroelectric-metal heterostructures become more interesting: indeed, the surface charge in a ferroelectric semiconductor plays the same role as the doping charge and it is, in principle, possible to modify the barrier characteristics from Schottky to ohmic without having to dope the material.[2,7] Because of this, the interest on ferroelectric resistive switching has increased in recent years.[8–18]

In order to take into account the polarization screening at the interfaces (or the so-called "dead-layer"),[19] one can consider the metal-ferroelectric junction to be a Schottky contact with the ferroelectric polarization modeled as a charged sheet at a certain distance of the physical interface with the electrode.[20,21] Assuming small (and non-overlapping) depletion layers, this model has given experimental Schottky barrier height (SBH) values in the range of $\varphi_o = 0.2$–0.9 eV for various ferroelectrics and orientations.[22–24] Classically, the "dead layer" (non-switchable interface layer) was assumed to be associated to defects in the film. But even in the case of an ideal, defect-free, ferroelectric layer, a "dead-layer" or series capacitance always exists due to imperfect screening. This can show up as electronic or ionic effects. Different ionic displacements at the interface could be as important as the electronic effects[25–27] or even give rise to a super-polarized layer ("negative dead layer").[28]

For "bad" metal electrodes (large screening lengths) and for ultrathin ferroelectric layers as those requested for tunnel-junctions,[29,30] the charge at the electrode may not be enough to screen

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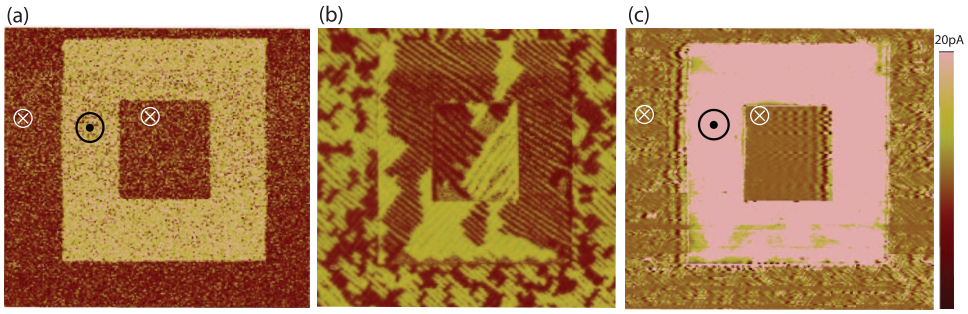

FIG. 1. (a) Out-of-plane piezo-force microscopy (PFM) phase image showing the as-grown down-polarized state (cross, outside dark frame). A square of polarization up (dot, light contrast) has been written. In a second inner square, the polarization has been switched back down. (b) In-plane phase map used (together with the in-plane amplitude map-not shown) to infer the types of domain walls. (c) Conductive-AFM (C-AFM) image of the same area. The applied bias voltage is 2.9 V. All areas are 20 μm $\times$ 20 μm.

the polarization and a large depolarization field may be present.[31–33] Following the Thomas-Fermi approach, Zhuralev *et al.*,[34] pointed out that using electrodes with different screening lengths would lead to changes of several orders of magnitude in the tunneling conductance through the ferroelectric upon polarization switching, (*giant electroresistance effect*).[31,34] Less discussed has been the way in which ferroelectric switching modifies the resistance through thicker layers (when direct tunneling is not possible) in case of incomplete screening at the electrodes. In this case of thicker layers, the average potential barrier height across the ferroelectric layer is not the determining parameter for conduction and resistive switching can be obtained with symmetric electrodes.[17,25]

Moreover, it is believed that the presence of vacancies and charged defects plays an important role determining conduction through the ferroelectric layers so that both polarization charges and defect charges need to be considered to understand the resistive switching phenomena in ferroelectrics.[8,10–15,35] However, there does not seem to be a clear understanding about the relative importance of the two. In order to shed light on the open problems discussed above, in the present letter, we artificially reverse the polarization, using piezo-AFM, and investigate the differences between the conduction through up-polarized domains and that previously reported for the down-polarized (as-grown) domains[36] of epitaxial $BiFeO_3$, a ferroelectric that is known to display remarkable resistive switching.[8,11,12]

$BiFeO_3$ thin films with thicknesses about 70 nm were grown by pulsed laser deposition on single-terminated (001)-$SrTiO_3$ substrates covered by a buffer electrode layer of $SrRuO_3$ with a thickness of 5 nm, as described in Ref. 36. The as-grown films were down-polarized (polarization towards the substrate), with only four of the eight pseudo-rhombohedral domains present.[37] This preferential poling is common in ferroelectrics[38] and could be due to an increased density of oxygen vacancies at the top surface, induced during growth. I–V curves as a function of temperature were measured locally using conducting atomic force microscopy (C-AFM) in air.

By applying a bias to the metallic tip of the atomic force microscope (in contact mode), it is possible to reverse the polarization of the $BiFeO_3$ layer from the as-grown down state to the up state. As a consequence, an increased conduction is observed in the up-polarized region, as reported in other bulk and thin film samples.[8,11,12] Figure 1 shows that the current through the ferroelectric can be increased by one order of magnitude upon polarization switching.

Figure 2(a) shows the temperature dependent I–V characteristics measured in the up-polarized domains. As it was done for the down-polarized domains and for the domain walls in Ref. 36, the different possible mechanisms for conduction through a semiconductor (i.e., Fowler-Nordheim tunneling, Poole-Frenkel emission, and Schottky-like emission, as well as space charge limited conduction) have been investigated. As in the case of the down-polarized domains,[36] we find that the I–V characteristics at high temperatures and large bias voltages are in good agreement with the Simmons-Richardson-Schottky (SRS) conduction model.[39] Indeed, this is the case for temperatures above 75 °C and voltages above 3.8 V (see linear be-

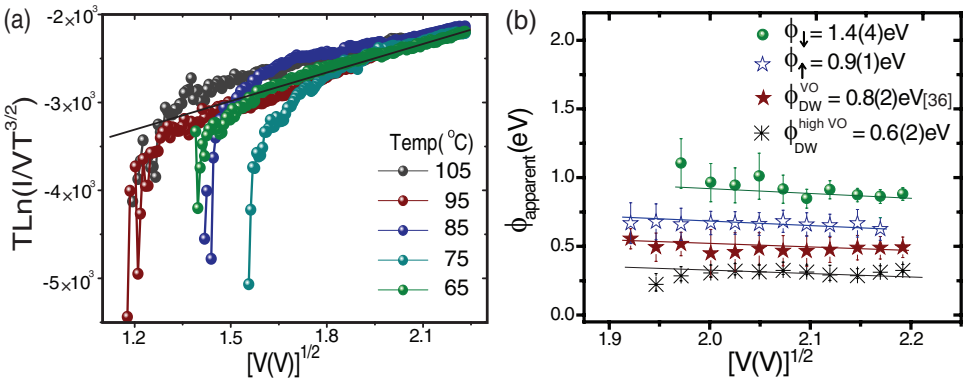

FIG. 2. (a) The IV curves corresponding to artificially switched up-domains are plotted in such a way that those data points that fulfill the Simmons-Richardson-Schottky equation[39] collapse on a single line (here SRS behavior takes place at high T and high V). From the slope of the SRS curve (dashed line), a value $\varepsilon_\infty = 6.1(3)$ is obtained. (b) When $\varphi_{\text{apparent}}$ (the argument in the SRS exponential-see main text) is plotted as a function of $V^{1/2}$, the intercept at the origin gives the (zero voltage) barrier heights. Data in Fig (a) are plotted as open blue stars. IV curves measured at the domain walls (DWs) in samples with higher oxygen vacancy content (see asterisks), also fulfill the SRS model. Data for down-polarized domains and for DWs with lower oxygen vacancy content (taken from Ref. 36) are presented for comparison purposes (green balls and red stars, respectively).

havior in Fig. 2(a)). SRS behavior is also found at lower temperatures but then in a narrower voltage regime (see 65 °C curve in the same figure). For V > 5 V, the current increases sharply because polarization switching starts taking place. We have, thus, restricted ourselves to V < 5 V for the subsequent analysis. According to the SRS equation,[39] the ordinate in Fig. 2(a) is the so-called "apparent barrier,"[22] $\varphi_{\text{apparent}} = \varphi_o - (e^3V/(4\pi\varepsilon_0\varepsilon_\infty d))^{1/2}$. In our case, the ordinates represent the "apparent effective *barrier*" modified both by polarization and screening charges. Such $\varphi_{\text{apparent}}$ is plotted in Fig. 2(b) as a function of $\sqrt{V}$ in the voltage regime that fulfills the SRS model (3.8 V < V < 4.8 V). From fitting those linear plots, a zero-voltage effective barrier of $\varphi_\uparrow = 0.9(1)$ eV has been obtained for the up-polarized domains. For comparison, in the same figure, we have plotted the data corresponding to down polarized domains (new data with improved statistics compared to Ref. 36), domain walls with lower oxygen vacancy content (data taken from Ref. 36), and domain walls with higher oxygen vacancy content.

Despite the limited voltage range accessible to our experiments, plausible optical dielectric permittivities can be extracted from the slopes of Fig. 2(b): $\varepsilon_\infty = 6.1(3)$, 6.5(2), and 7.5(1), for up-polarized domains, domain walls with intermediate oxygen vacancy content and domain walls with high vacancy content, respectively. They are in very good agreement with the value of $\varepsilon_\infty = 6.25$ reported for $BiFeO_3$.[21] Only for the down-polarized domains (data from Ref. 36), the linear regression produces a significantly larger slope, giving rise to a less physical value of $\varepsilon_\infty = 1.8(3)$.[36] The increased slope produces a zero-voltage barrier of $\varphi_\downarrow = 2.1(3)$ eV. It is to be noted that the down-polarized domain state is the highly resistive as-grown state with room temperature leakage current smaller than 1 pA, leading to a low signal-to-noise ratio. Because of this, only above 115 °C proper IV curves are obtained[36] producing a large uncertainty in the determination of the barrier height. Since there is no physical reason to expect a different value of ε_∞ in this case, we have fixed the slope of $\varphi_{\text{apparent}}$ in down-polarized domains (green balls in Fig. 2(b)) to that corresponding to $BiFeO_3$, $\varepsilon_\infty = 6.25$[21] (green line in Fig. 2(b)). This gives rise to a $\varphi_\downarrow = 1.4(4)$ eV and, therefore, to a difference of $\Delta\varphi = 0.5(5)$ eV between up-polarized and down-polarized domains, which is comparable to that obtained by directly measuring the work function in vacuum.[16] This result is consistent with earlier reports by Pintilie *et al.*, showing that the height of the electronic barrier determines the current through a thick $BiFeO_3$ films when using macroscopic electrodes.[22] Also, in agreement with the n-doped character of the $BifeO_3$ layer (due to oxygen vacancies), the observed conduction corresponds to electrons injected from the top Cr-electrode.

The high conduction state in the up-polarized domains persists at room temperature for (at least) weeks after poling. However, increasing the temperature to (and above) 180 °C, the up-polarized

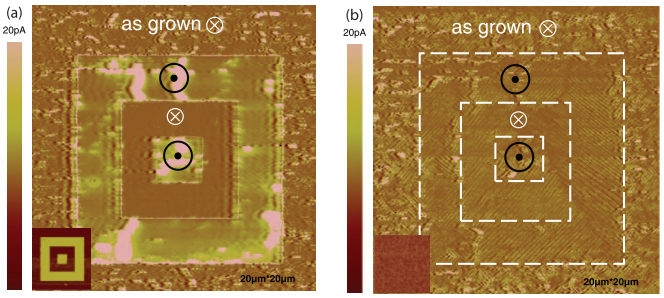

FIG. 3. Conduction maps of the sample surface after writing an up-polarized square, an inner down-polarized square and still a smaller up-polarized square in the center. (a) The encircled dots and crosses denote the regions with polarization up and down, respectively. The imaging voltage used was 2.4V, smaller than that of 1 in order to avoid saturation of the TUNA amplifier. The out-of-phase PFM image is also shown as an inset in the lower left corner. This image is stable for weeks at room temperature. (b) shows the conduction map under the same DC voltage bias after annealing the sample for 1 h at 200 °C.

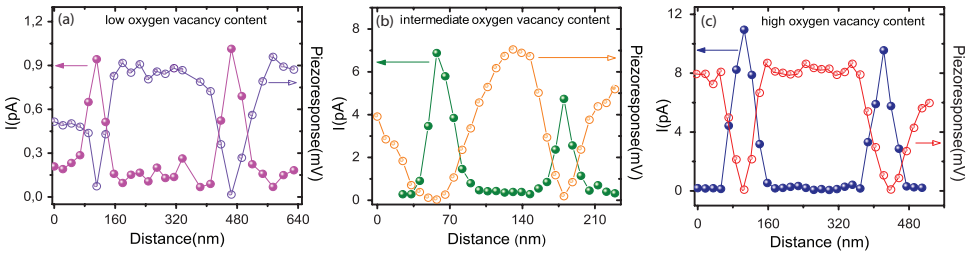

FIG. 4. Line scan of both piezoresponse amplitude (open symbols) and current (filled circles) across one down-polarized domain with two adjacent domain walls in three samples grown under identical conditions but subjected to different annealing conditions: (a) annealing pressure of $PO_2 = 300$ mbar and a cooling rate of 1 °C/min, (b) $PO_2 = 100$mbar and rate of 3 °C/min, and (c) $PO_2 = 100$ mbar and rate of 40 °C/min.

regions switch back within 1 h and the low conduction state is recovered (see Fig. 3). This could point to adsorbates as screening charges of the up-domains, as it will be discussed below (our samples have no permanent top electrode since we use as such the metallic tip of the AFM set-up). As the screening adsorbates are removed at high temperatures, the up-polar domains become unstable, the films switch back to the as-grown state (inset in Fig. 3(b)) and the current decreases to the highly resistive state.

In previous works, we have also qualitatively shown the importance of oxygen vacancies and their strong influence on the change of conduction at the domain walls (DWs).[36] At 71° DWs, electronic effective barriers of $\varphi_{DW} = 0.8(2)$ eV[36] and $\varphi_{DW} = 0.6(2)$ eV (this work), and thus different conductivities, have been observed in samples with lower (standard annealing) and higher oxygen content, respectively, as shown in Fig. 2(b). However, the influence of the oxygen vacancy content on the conduction through the domains themselves has not been reported. In Fig. 4, we compare three as-grown (down-polarized) samples grown under the same conditions but cooled down with different rates and/or O_2 pressure. We observe that while the current at the domain walls can be increased by a factor of 10 simply by increasing the density of oxygen vacancies, the current through the domains remains unchanged. This is consistent with the model of positively charged oxygen vacancies migrating towards the walls and reducing the electronic barrier locally around them.[36] It is also consistent with the screening of the polarization charges at the top surface in the down-domains by oxygen vacancies, such that additional vacancies do not have significant effect on the conduction through domains.

To a first approximation, the difference between the work function of the metal and the electron affinity of $BiFeO_3$ gives rise to a nominal Schottky barrier height,[40,41] of $\varphi_o = 1.4$ eV for the top Co/Cr-$BiFeO_3$ interface.[42] Apart from other smaller corrections,[40] this value assumes non-polar $BiFeO_3$ and a clean metal-semiconductor interface, both conditions not being satisfied in our case:

the bound surface charges associated to a polarization pointing down inside the ferroelectric domains are expected to increase the barrier height for electrons injected from the Cr electrode, as indeed observed. In addition, measurements in air allow for adsorbates to act as screening charges and they also contribute to changes in the electronic potential. Here we refer to this as the *effective barrier height* φ_{eff}, to emphasize the fact that we do not deal with an ideal metal-semiconductor Schottky barrier.

Interestingly, while admitting the sources of error discussed above, the value $\varphi_{\downarrow} = 1.4(4)$ eV obtained for the down domains (Fig. 2(b)) agrees with the nominal φ_o of a non-polar $BiFeO_3$/Cr interface. This could indicate that the bound surface polarization charges are neutralized by the oxygen vacancies created during the growth (note that no adsorbates are present in the growth vacuum chamber). An excess of oxygen vacancies close to the top surface in $BiFeO_3$ has been earlier reported.[24] This scenario can also explain why the as-grown samples are self-polarized with down polarization.

For the down-state, in the presence of bound polar charges, as well as screening charges, the electronic barrier can be written as $\varphi_{\downarrow} = \varphi_o + \Delta\varphi_P - \Delta\varphi_{\downarrow scr} = 1.4(4)$ eV (measured value), implying that $\Delta\varphi_P - \Delta\varphi_{\downarrow scr} = 0.0(4)$ eV (that is, consistent with full screening within a large experimental error). For the up polarized state, the barrier height will then become $\varphi_{\uparrow} = \varphi_o - \Delta\varphi_P + \Delta\varphi_{\uparrow scr} = 0.9(1)$ eV (measured value), thus $\Delta\varphi_P - \Delta\varphi_{\uparrow scr} = 0.5(1)$ eV. The large experimental error does not permit to discern between two possible scenarios: (1) the screening mechanism is the same in the two polarization states ($\Delta\varphi_{\uparrow scr} = \Delta\varphi_{\downarrow scr}$), what gives rise to a contribution to the barrier height due to the polar charges of $\Delta\varphi_P = 0.25(2)$ eV and (2) the screening mechanisms are different, with $\Delta\varphi_{\uparrow scr} - \Delta\varphi_{\downarrow scr} = 0.5(5)$ eV. Given the asymmetry of the film surfaces, the first scenario is unlikely, since the electronic charges that are able to screen the bound polar charges at the interface with the electrode in the down–state are not available at the top surface in the up-state. There must be, thus, a different screening mechanism for the two polar states. For the up-polarized domains this mechanism is less efficient than that of the down-state (as-grown oxygen vacancies), giving rise to the observed lowering the electron barrier in the up-domains by 0.5(5) eV. As mentioned, the instability of the up-polarized state upon increasing temperature demonstrated in Fig. 3, points to adsorbates as the most likely screening charges in the up-state.

All the above implies that experiments with samples having an extended top electrode will prevent adsorbates on the top ferroelectric surface and would lead to different resistance changes than those observed here. Moreover, one can expect that diffusion of oxygen vacancies and other charged defects inside the ferroelectric is easier in poli-crystalline samples or films showing columnar growth than in fully epitaxial films. Therefore, the exact interplay between the polarization and screening charges varies considerably with the sample morphology and device geometry, explaining the different behaviors reported in the literature.[8,15] In addition, since this is mainly an interface effect, the type of structural interfaces and twinning is of crucial importance to determine the dominant type of conduction. The films discussed exhibit only in-plane twinning, that is, the (001) planes of the film are parallel to those of the substrate,[37] which should be favorable for conduction.[22,37] It is also worth to mention that the in-plane twinning gives rise to a more clamped structure with larger coercive fields than when the interfaces are buckled by out-of-plane twinning.[22,43] In fact, we can apply up to 5 V without switching the polarization or inducing domain movement, which allows to increase the current values up to 15 pA at room temperature. Given that the tip diameter, analyzed by Scanning Electron Microscopy, is in between 50 nm (new tip) and 100 nm (used tip), the observed current densities can be as high as 0.5 A/cm^2 at room temperature. Even larger current values of 5.4 A/cm^2 have been obtained in different $BiFeO_3$ films with a more grainy microstructure.[8]

In summary, we have measured a change in the barrier height for electron injection, when switching the polarization of $BiFeO_3$ film from (as-grown) down-polarized to up-polarized, which explains the previously observed large changes in resistance for bias voltages applied parallel or antiparallel to the polarization direction. We argue that screening charges play a very important role and that they are of different nature in the two polar states.

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